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(54) Title of the invention

Optical resin molding

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(72) Inventor: Niwano Masahiro

Sumitomo Chemical Co. Ltd.

Ehime-ken, Niihama-shi, Sobiraki-cho 5-1

(72) Inventor: Manabe Kenji

Sumitomo Chemical Co. Ltd.

Ehime-ken, Niihama-shi, Sobiraki-cho 5-1

(72) Inventor: Moriya Masahiko

Sumitomo Chemical Co. Ltd.

Osaka-fu, Takatsuki-shi, Tsukahara 2-10-1

(71) Applicant(s): Sumitomo Chemical Co. Ltd.

Osaka-fu, Osaka-shi, Higashi-ku, Kitahama 5-15

(74) Representative: Patent Attorney Moriishi Mitsuhiro (and one other person)

1. Title of the invention

Optical resin molding

2. Scope of the patent

- (1) In optical resin molding formed from a polymer portion comprising mainly of an aromatic vinyl monomer and polyphenylene ether portion, when the melt flow of the polymer portion comprising mainly of the aromatic vinyl monomer (conforming to ASTM D1238 at 230°C, 3.8 kg load and g/10 min) is represented as (MFR)_{ps}, the polymerization rate of the aromatic vinyl monomer unit in the said polymer portion is represented as W_{ps}, the intrinsic viscosity (measured and calculated in a chloroform solvent at 25°C using the Ubbelohde viscometer) of the polyphenylene ether portion is represented as (η)_{PPE} and the polymerization rate of the polyphenylene ether portion in the optical resin mold is represented as W_{PPE}, the (η)_{PPE} had a value of 0.3 ~ 0.7 and a general formula as shown below.

$$-1.4 \quad \frac{1}{W_{ps}} \left(1.13 + 0.07 \log_{10} \frac{20}{(MFR)_{ps}} \right) = Y$$

(7)

PPE

$$Y - 2 \leq 100 \cdot W_{PPE} \leq Y + 2$$

3. Detailed description of the invention

(Fields of industrial usage)

This invention is concerned with optical resin molding.

It is concerned with the molding of optical elements for optical disk substrates, lenses, prisms etc. using specific resins.

(Conventional techniques)

Using the laser optic spot, the record information that has been engraved on minute unevenness on the disk substrate is detected and the regeneration of the information record of high density is carried out by regenerating the image or sound or by changing the optical properties of the record engraved on the substrate surface. It is this regeneration of the record that has recently drawn attention.

The disk substrate that is used in such a form of record regeneration requires that it have transparency, the dimensional stability be good, it is optically homogenous, the double refraction be small and so on.

With resin material being used as the disk substrate and even though large quantities of duplicated substrates can be molded at a low cost, in many cases molecular orientation occurs in the liquefaction as well as cooling process of the resin at the time of molding of the disk substrate. It is widely known that double refraction occurs which is said to be a fatal defect.

Since it is difficult to avoid molecular orientation during molding, especially the one carried out through injection molding, at present the only polymer having methyl

methacrylate as the main constituent is available as the resin material with low optical anisotropy that would be suitable for the optical disk substrate mold.

However, when the polymer having methyl methacrylate as the main constituent is used in the substrate, because of its highly hygroscopic nature, the dimensional stability becomes bad, it warps under high humidity and torsion also occurs.

This defect has been mentioned in the Nikkei Electronics journal (07 June 1982 issue; Page 133). For this reason, an aromatic polycarbonate resin that has a low hygroscopic value is used as the material in audio compact disks.

Since the aromatic polycarbonate resin includes an aromatic ring having a high anisotropy in its main chain, it is difficult to reduce the double refraction of the mold substrate. Although studies on reduction of molecular weight and mold formation conditions are being conducted, due to the fact that the double refractivity originates in that raw material, it is difficult to uniformly stabilize and produce a substrate with a low double refraction. Therefore what needs to be improved is the low double refractivity and production of a substrate through injection molding having a diameter larger than that of the audio compact disk.

For improving the dimensional stability, which is the drawback in the polymer having methyl methacrylate as the main constituent, a co-polymer substance comprising methyl methacrylate and an aromatic vinyl monomer has been proposed in Patent Notifications Sho 57-33446, Sho 57-162135 and Sho 58-88843.

However, in co-polymerization with a vinyl monomer having an aromatic ring, double refraction occurs easily and therefore cannot be put to practical use.

For the disk substrate obtained not only by regeneration of information but also by registration, although a much more superior double refractivity and dimensional stability is required, yet no resin material has been discovered that fully satisfies these requirements.

Even in the optical elements in lenses, prisms etc., resin material such as methacrylate resins and others have been used until now but what is being demanded is a resin material having a much lower double refraction, excellent heat resistance, mechanical strength and dimensional stability.

In US Patent Notification 4,373,065, it has been reported that the resin material has an exactly opposite optical anisotropy. Here, two types of polymer that melt completely are mixed in a composition that exactly negates that optical anisotropy and it results in an optically recorded element which is formed from an optically isotropic resin in which the double refractivity becomes practically zero.

In the said Patent Notification it has also been mentioned that in the system of using polyphenylene ether and polystyrene as the polymers having an exactly opposite optical anisotropy, even if stress is applied on the film that is made from the composition of the mixture that exactly negates that optical anisotropy, double refraction does occur. In other words, when a polymer compound in a solid state is stretched, it was seen that double refraction did not occur.

(The problems that this invention seeks to solve)

The US Patent Notification 4,373,065 mentioned above does not mention as to how the double refractivity of the optical material becomes remarkably small when the

said optical material for the optical disk substrate is produced through injection molding using a polymer compound as the base material.

The optical anisotropy exists in the solid state. When two types of polymer that melt completely are mixed in a composition, the optical anisotropy in that solid state is seen to exactly negate that of the mixture composition. When the substrate was molded through injection molding, a recent production method, it was found that the double refraction of the mold obtained was not necessarily small.

In other words, when an optical raw material for the optical card substrate is to be prepared through injection molding using the polymer compound as the raw material, the optical material having a small double refraction cannot be obtained merely by singularly matching the solid state optical anisotropy for individual polymers to the composition in which it has been thought of.

In recent years, tests have been conducted to produce disk substrates for optomagnetic disks made of plastic on which erasure and rewriting would be possible.

Thus, when the recorded information on the optic disk in the optomagnetic form is read, the polarized laser rays are linked to the focus in the record medium in the lens and depending on the effect that is reflected, the information is read by detecting the circling of the scantily present polarized light in the laser rays. Thus, even for slanted incident light rays, it is necessary to use optical disk substrates where it is difficult for double refraction to take place.

Since the medium is heated due to laser rays during writing, it is necessary to have optical disk substrates having high heat-resistance.

This invention is cognizant of these problems and provides for an optical element that has a low double refraction even for the ones produced through injection molding, compression molding and other such methods, a low double refraction for the slanted incident light rays and also has a high heat-resistance, a good balance of mechanical strength and an excellent dimensional stability.

(Procedure for solving these problems)

In the optical resin mold resulting from a polymer portion comprising mainly of an aromatic vinyl monomer and polyphenylene ether portion, when the melt flow of the polymer portion comprising mainly of the aromatic vinyl monomer (conforming to ASTM D1238 at 230°C, 3.8 kg load and g10 min) is represented as (MFR)_{ps}, the polymerization rate of the aromatic vinyl monomer unit in the said polymer portion is represented as W_{ps}, the intrinsic viscosity (measured and calculated in a chloroform solvent at 25°C using the Ubbelohde viscometer) of the polyphenylene ether portion is represented as (η)_{PPE} and the polymerization rate of the polyphenylene ether portion in the optical resin mold is represented as W_{PPE}, the (η)_{PPE} had a value of 0.3 ~ 0.7 and a general formula as shown below.

$$-1.4 \quad \frac{1}{W_{ps}} \left(1.13 + 0.07 \log_{10} \frac{20}{(MFR)_{ps}} \right) = Y$$

PPV

$$Y - 2 \leq 100 \cdot W_{PPE} \leq Y + 2$$

The optical resin mold in this invention is a mixture of a polymer having an aromatic vinyl monomer unit as the main constituent and polyphenylene ether or it is a block co-polymer or graft co-polymer resulting from the polymer portion of both the compounds or else it is a resultant mixture of all of these.

The polymer having the aromatic vinyl monomer unit as the main constituent mentioned in this invention is the aromatic vinyl monomer-independent polymer and also it is the co-polymer which has more than 50 weight % of an aromatic vinyl monomer unit. Styrene, α -methyl styrene, m-methyl styrene, p-methyl styrene, o-chlorostyrene, m-chlorostyrene, p-chlorostyrene, m-bromostyrene, p-bromostyrene etc. are examples of aromatic vinyl monomers, out of which styrene is the most desirable.

Examples of monomers that co-polymerize with aromatic vinyl monomers are unsaturated nitril class compounds like acrylonitril and methacrylonitril; alkyl ester methacrylate class compounds like methyl methacrylate, n-propyl methacrylate, isopropyl methacrylate, n-butyl methacrylate, cyclohexyl methacrylate; alkyl acrylate class compounds like methyl acrylate, ethyl acrylate, propyl acrylate, butyl acrylate etc. There are also other compounds like methacrylic acid, acrylic acid, maleic anhydride, citraconic anhydride, N-methyl maleimide, N-phenyl maleimide etc. that can be used

The polymers that co-polymerize with these compounds can each be used either independently or as a mixture. It is better if the combination and regulation of the usage ratio of the co-polymer from the aromatic vinyl monomer as well as the resin material resulting from the aromatic vinyl monomer and polyphenylene ether are carried out without any hindrance.

It is desirable that the proportion of the aromatic vinyl monomer in the monomer mixture be more than 50 weight %. If the aromatic vinyl monomer is less than 50 weight % then the hygroscopic level of the resin becomes high, which is not desirable.

The Melt Flow Rate (MFR)_{ps} of the polymer having the aromatic vinyl monomer unit as the main constituent conforms to ASTM D1238 and the melt flow rate at 230°C and 3.8 kg. load is represented as g/10 min.

The melt flow rate is given to be 0.5 ~ 200; however, the desirable range is 2 ~ 100.

If it exceeds 200, then the mechanical strength declines, which is not desirable and if it is less than 0.5 then reduction of double refraction becomes difficult, which is also not desirable.

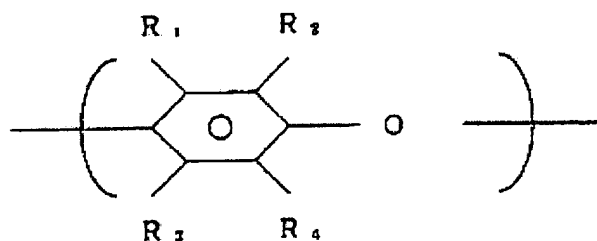
The method of production of the polymer that has the aromatic vinyl monomer unit as the main constituent could be any of the methods that use a radical initiator such as block polymerization, suspension polymerization, emulsion polymerization or solvent polymerization. However keeping in mind the objective of obtaining a polymer with good productivity and having fewer impurities mixed in, block polymerization or suspension polymerization is desirable.

Peroxides like lauryl peroxide, benzoyl peroxide, di-tert-butyl peroxide, di-cumyl peroxide and azo compounds like 2,2'-azobis-isobutyronitrile, 1,1'-azobis (1-cyclohexane carbonylnitrile) can be used as radical initiators.

To control the molecular weight, chain-transfer agents such as tert-butyl, n-butyl, n-octyl, n-dodecyl and tert-dodecyl mercaptane may be added.

Polymerization is generally carried out in a temperature range of 50 ~ 150°C.

The polyphenylene ether mentioned in this invention is represented as the general form and is a polymer that has repeating units.



(Here, R₁, R₂, R₃, R₄ indicates a hydrogen, halogen or a hydrogen carbide radical.)

The said polyphenylene ether is a polymer that has been polymerized from a phenol class monomer through oxide coupling. Polyphenylene ether is a compound that can be easily manufactured through known methods (Patent Notifications Sho 36-18692 and Sho 47-36518) using a copper group or manganese group catalyst.

Definite examples of polyphenylene ether are compounds such as poly(2, 6-dimethyl-1,4-phenylene)ether, poly(2-methyl-6-ethyl-1,4-phenylene)ether, poly(2-methyl-6-propyl-1,4-phenylene)ether, poly(2, 6-dipropyl-1,4-phenylene)ether, poly(2-methyl-6-bromo-1,4-phenylene)ether etc., out of which poly(2,6-dimethyl-1, 4-phenylene)ether is preferred.

This polyphenylene ether is generally used as engineering plastic but a compound with a much lower molecular weight is suitable.

The average molecular weight of polyphenylene ether is shown at the intrinsic viscosity of the polymer (η)_{PPE} (measured and calculated in chloroform solution at 25°C) and is 0.1 ~ 1.0; however the desired molecular weight is 0.3 ~ 0.7.

Narrowing it down, a molecular weight of 0.3 ~ 0.45 is desirable and a weight of 0.35 ~ 0.42 would be highly desirable.

If the molecular weight is less than 0.3, then the mechanical strength of the optical resin mold becomes small.

Further, to make the double refraction and also the double refraction of the slanting incident light rays much smaller, a molecular weight below 0.45 is desirable.

It is thought that this is because the ease in orientation of the polymer portion that has the aromatic vinyl monomer as the main constituent and the polyphenylene ether respectively and/or the rise in speed of the polymer in the metal pattern differs when the optical element is produced through injection molding.

In other words, for a polyphenylene ether that has a high intrinsic viscosity, orientation occurs easily and the orientation is fixed during molding; and then depending on the average molecular weight used, the orientation of the polyphenylene ether portion in the resin becomes difficult and the rise in speed too becomes faster. From this it is thought that the optical resin mold having a small double refraction as also a small double refraction for the slanting incident light can be obtained from a much wider range of resin compositions and under much wider injection molding conditions.

To obtain the resin material that will be used in optical resin molding by mixing a polymer that has the aromatic vinyl monomer as the main constituent and polyphenylene ether, methods such as fusion mixing or solution mixing are suitable.

Fusion mixing is carried out at a temperature higher than that of the fusion temperature of the polyphenylene ether and under high shearing, for which mixing machines such as an extruder, Banbury mixer, kneader-blender, heat rolls etc. are used.

The degree of fusion should be to the extent that both the polymers are mutually dispersed and mixed upto approx 1 μ and further on they are mixed upto the molecular scale.

Whether the mixture state has reached the molecular level or not can be judged easily when the glass transition temperature of the mixture is a constant.

In order to obtain a very satisfactory mixture state, methods such as raising the mixing temperature, prolonging the mixing time and raising the shearing force are adopted.

Further in fusion mixing, to make the mixing easy by lowering the mixing temperature of both the polymers, small quantities of an organic solvent can be used as a plasticizer.

As for the organic solvent, the one that is used in the solvent mixing method mentioned in subsequent pages can be used here. After the mixing process is completed, it is better if the used organic solvent is removed through evaporation.

In solvent mixing, both the polymers are dissolved in the organic solvent and at least 1 weight % of the solvent is produced. After getting a uniform mixture through stirring, the organic solvent is removed through evaporation; alternatively, it is also possible to add a low-solvent in both the polymers to obtain a uniform mixture and then precipitate the polymers that have been mixed.

The suitable organic solvents are chloroform, methylene chloride, ethylene chloride, toluene, benzene, chlorobenzene etc. Low-solvents are compounds such as methanol, ethanol, propyl alcohol, n-hexane, n-pentane etc.

The block co-polymer or graft co-polymer formed from the polymer portion having an aromatic vinyl monomer unit as the main constituent and the polyphenylene ether portion is obtained by polymerizing the polymer on one side with the monomer on the other side.

In specific terms, as per the methods mentioned in Patent Notifications Sho 42-22069, Sho 47-1210, Sho 47-47862 and Sho 52-38596, the monomer that has the aromatic vinyl monomer as the main constituent is polymerized in the presence of polyphenylene ether. Alternatively, the graft polymer or block co-polymer can be produced by polymerizing a phenol class monomer through oxide coupling in the presence of a polymer that has an aromatic vinyl monomer unit as the main constituent.

In order to sufficiently minimize the double refraction for the optical resin mold not only with respect to the vertically incident light rays but also for the slanting incident light rays, it is necessary to have a composition that fits the general formula.

Further, it is desirable that the weight proportion W_{PPE} of the polyphenylene ether portion in the general formula be in excess of 0.4 and below 0.7.

If the weight proportion is below 0.4 then the heat-resistance will not be enough and if it is above 0.7 then staining develops to a large extent and mold conditions also develop rigorously. Therefore, a weight proportion outside of this range is not desirable.

The said resin mold in this invention, besides being used in general optical disk substrates can also be used in optomagnetic disk substrates, all types of lenses, prisms etc..

When the said resin mold is considered for optical disk substrates, then semiconductor laser optics comes to mind.

Hence, it is desirable that the light transmission rate for an 800 nm wavelength be more than 75% for a material having a thickness of 1.2 mm.

In this invention, the molding methods for preparation of the optical resin mold could be any of the methods like injection molding, compression molding, injection compression molding or others. Out of these methods, the effect of this invention is said to be remarkable for the method in which a comparatively high double refraction is produced vis-à-vis the mold. From this and also from the productivity point of view, the injection molding method is said to be the most desirable.

In the injection molding method mentioned in this invention, the process involves applying heat to a closed metal pattern cavity followed by injecting the resin in the fluid state into it, cold solidification and finally production of the mold item

The process of applying heat within the metal pattern cavity could be by vacuum suction method or injection compression method where the metal cavity volume is reduced. Here, both the methods may be used together.

For the optical resin mold produced through the injection molding method mentioned in this invention, it is desirable that the injection molding of the melted plasticized resin is done in a temperature range of 270°C-350°C; however a temperature of 300°C to 340°C would be desirable.

The resin temperature mentioned here is the external heat of the heater etc. in the injection-molding machine and it is the temperature of the resin in the injection cylinder that has undergone plasticized fusion due to shearing heat generation through screw rotation.

If the temperature of the resin is far below 270°C, then the measurement of double refraction of the optical disk substrate will be more than 20 nm and would therefore be unsuitable as an optical disk substrate. And again, if the temperature exceeds 350°C, then poor results such as dissolution of the resin, yellowing, silvering etc occur and the pit error of the optical disk substrate increases remarkably.

In the injection molding method of this invention, it is desirable that the temperature of the metal pattern cavity is maintained between 50°C and 124°C; however a more desirable range would be between 80°C to 120°C.

The metal pattern temperature mentioned here is the surface temperature of the metal cavity immediately before the injection.

If the metal temperature is below 50°C, then the transference of the minute guide grooves engraved on the surface of the metal pattern becomes bad. And again, if the metal temperature exceeds 140°C, then the mold release from the metal becomes bad, which is not desirable.

In the injection molding method of this invention, it is desirable that molding is carried out in a dwell time ranging from 0.2 seconds to 3 seconds. However, a more desirable range would be above 0.3 seconds and below 0.2 seconds.

The dwell time mentioned here is the time taken for replenishing the resin in the metal cavity.

If the dwell time is below 0.2 seconds, then silvering occurs and when it is used as an optical disk, the pit error increases remarkably. If the time exceeds 3 seconds, then the double refraction of the optical disk substrate goes above 20 nm, which is not desirable.

(Application examples)

This invention is explained below in detail through application examples. The invention however, is not restricted only to these examples.

Both the division and percentage in these Application Examples are the weight standards.

The properties of the substances shown in the Application Examples have been measured through the methods given below.

- *Double refraction:* The retardation was measured through the Senarmont Compensator method using a polarizing microscope and laser rays of wavelength 546 nm.
- *Suction rate:* The equilibrium suction rate in 60°C-distilled water was measured and it was based on ASTM D-570.
- *Light rays transmission rate:* The light transmission rate for a sample thickness of 1.2 mm was measured through an auto-spectrophotometer (Hitachi-make; Model 330) at 800 nm.
- *Bending property:* The bending property measurement was based on ASTM D-570.
- *Heat resistance:* Using a 5 mm x 5 mm x 3 mm test piece, the glass transition temperature was measured through the Line Expansion Coefficient method.
- *Viscosity limit of the polymer:* The viscosity limit of the polymer was measured and calculated in a chloroform solvent at 25°C using the Ubbelohde viscometer.

- *Melt flow rate:* The melt flow rate was measured under a temperature of 230°C and 3.8 kg load conforming to ASTM D-1238.
- Kneading and pelleting were carried out using a bi-axial extruder. (Japan Seiko Co. Ltd-make; Model TEX30-30BW-2V).
- The injection-molding machine used in the Application Examples was a Sumitomo Heavy Machinery Industries-make Neomatt Model 150/75 (75 tons); the metal pattern had a disk metal pattern with a mold diameter of 120 mm and a thickness of 1.2 mm.

Application Examples 1, Comparative Examples 1 ~ 3

In accordance with the method given in Application Example 2, No.9 in Patent Notification Sho 47-36518, 2, 6-xylenol was polymerized using manganese chloride and ethanolamine as a catalyst from which poly(2, 6-dimethyl-1, 4-phenylene)ether having an intrinsic viscosity of 0.40 (in chloroform at 25°C) was formed.

This polyphenylene ether and polystyrene resin (Sumitomo Chemical Industries make Esbrite[®] 8-62, MFR 15) was mixed and compounded as per the proportion shown in Table 1. After kneading and granulating it through an extruder, injection molding was carried out at a cylinder temperature of 320°C and metal pattern temperature of 105°C further to which a disk having a diameter of 120 mm and a thickness of 1.2 mm was obtained.

The properties were evaluated and the results have been collated and shown in Table 1.

Table 1

		Application	Comparative	Comparative	Comparative
		Example 1	Example 1	Example 2	Example 3
Composition (%)	Polyphenylene ether	50	40	45	55
	Polystyrene	50	60	55	45
Light transmission rate (%)		86	88	87	86
Double refraction (nm)		+ 2	- 71	- 35	+ 40
Suction rate (%)		0.1	0.1	0.1	0.1
Glass transition temperature (°C)		149	142	145	154

Application Examples 2 ~ 5, Comparative Examples 4 ~ 8

As per the composition ratio shown in Table 2, poly(2, 6-dimethyl-1, 4-phenylene)ether having an intrinsic viscosity (in chloroform solvent at 25°C) as shown in Table 2 and polystyrene resin (Sumitomo Chemical Industries make Esbrite[®] 4-62 A, MFR 24) were kneaded and pelletized. This resin was injection-molded at a resin temperature of 320°C; a metal pattern temperature of 100°C and an injection molding time of 1 second and an optical disk substrate was produced.

The double refraction of the vertically incident light rays as well as the 30° slanted incident light rays in the optical disk obtained is shown in Table 2.

The measurement positions have been taken from the center of the optical disk substrate towards the radius.

Further, the bending properties and heat-resistance of the substrate obtained by press-molding the said pelletized resin at 270°C has been shown in Table 2.

Double refraction (nm)				Bending properties			Heat resistance
Vertically incident rays		30° slanted incident rays		Strength	Modulus of elasticity	Maximum distortion	° C
		25 mm	40 mm	55 mm	Kg/cd	%	
40 mm	+ 1	+ 2	+ 3	+ 1	+ 2	+ 2	146
	- 2	- 2	0	- 1	- 1	- 1	152
	+ 1	+ 1	+ 1	0	720	3.0	156
	- 1	- 1	- 1	- 2	400	1.5	153
	- 17	- 18	- 18	- 19	1000	3.4	140
	+ 16	+ 15	+ 20	+ 18	1150	4.4	151
	+ 11	+ 12	+ 12	+ 12	1090	3.6	157
	- 10	- 10	- 10	- 11	700	2.9	149
	+ 6	+ 6	+ 7	+ 6	430	1.7	159

The absolute value of the double refraction was more than 100 nm and the distribution unevenness was also large.

Comparative Example 11

Using bisphenol A as the base material and methylene chloride as the solvent, phosgene was blown according to the usual method and interface condensation was carried out. A polycarbonate resin with an average molecular weight of approx. 15, 000 was obtained.

Here, t-butyl phenol was used to regulate the molecular weight.

The powder resin obtained was then granulated through an extruder and injection molding was carried out under similar conditions as that in Application Example 1.

However, the double refraction of the disk obtained was more than +100 nm large.

The molding was carried out at a cylinder temperature of 340°C.

The double refraction of the disk was as large as 100 nm, the suction rate was 0.45 % and the glass transition temperature was 136°C.

At one point, the molding temperature was raised and the mold was tested. It was found that when the temperature exceeded 340°C, it was accompanied by thermal decomposition of the resin due to which molding became difficult.

Effect of this invention

Even though the optical resin mold of this invention has a low water absorptivity and good dimensional stability, yet it cannot be used because of the high double

refraction. The polystyrene group resin has not only become one of the raw materials that can be used but with this, it is also possible to obtain efficiency along with good heat resistance and balance of mechanical strength.

In particular, when a specific polyphenylene ether having a comparatively low intrinsic viscosity is used, the double refraction is small and the heretofore unknown double refraction of the slanted incident ray is also small. Besides this, a wider than usual composition can be obtained and because of this it has become possible to have a high heat-resistance in the compound.

In the method of production of an optical resin mold, it is easy for double refraction to take place. Even in the case of mold production through injection molding, it is said to have an advantage in that it is possible to have a mold with a low double refraction.

The optical resin mold of this invention is present in optical disk substrates, lenses, prisms etc. and even within that it is suitable when light rays having a specific wavelength are used.

In addition to what has been mentioned above, the double refraction for the slanted incident light rays is small and the mold has high heat resistance. Due to this, the general optical disk substrates are much more suited than they were before and they are also suited for use in optomagnetic disk substrates.